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Crystal structure refinement of a sepiolite/indigo Maya Blue pigment using molecular modelling and synchrotron diffraction

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Maya Blue is an artificial pigment used in Pre-Columbian America, renowned for its chemical stability. The pigment can be considered a precursor of modern inclusion compounds as a hosting microporous clay (palygorskite or sepiolite) shelters the guest indigo dye (≤2 wt%) within its micro-channels. While most papers on Maya Blue are focused on the interaction between indigo and palygorskite, this study describes the pigment structural features when sepiolite is the host structure. Synchrotron X-ray powder diffraction patterns were collected on both pristine sepiolite and sepiolite + indigo (2 wt%) pigment. The pigment structure was investigated with the Rietveld method, basing on both molecular mechanics and the refined structure of sepiolite. The evidence obtained shows that: (i) indigo molecules, encapsulated within the micro-tunnels, stay close to a TOT strip in order to receive H-bonds from the structural OH_2; (ii) there is no evidence for direct metal-oxygen bonds between the sepiolite Mg and the indigo C=O groups, as the applied heating (≤190 °C) does not remove structural OH_2; (iii) the indigo molecule is affected by 4-fold disorder, as it occupies only one of four partially superposed equivalent sites; (iv) indigo and the zeolitic H_2O compete to occupy the channels; refined occupancies showed that the dye fills 27 vol% of the channels whereas 73 vol% is occupied by H_2O. Calculated indigo weight % (1.9) is in close agreement with experimental data; (v) indigo encapsulation modifies

zeolitic H_2O sites, increasing the number and strength of mutual hydrogen bonds; (vi) difference-Fourier maps computed removing indigo contribution confirmed the position of the molecule inside the channels.

Key-words: sepiolite, indigo, Maya Blue pigment, molecular mechanics, Rietveld method.

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